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(57) Abstract

The invention relates to a method of reducing phytotoxicity to crop plants caused by at least one member of the group consisting of a 4-benzoylisoxazole herbicide and a 2-cyano-1,3-dione herbicide which comprises applying to the locus of the crop plant, the crop or crop plant seed, an antidotally effective amount of at least one sulfonyluera herbicide.

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New Herbicidal Mixtures

Field of the invention.

This invention relates to a method of safening herbicidal 4-benzoylisoxazoles and 2-cyano-1,3-diones by sulfonylurea herbicides, and to compositions containing the same.

Background of the invention.

It is known that many herbicides injure crop plants at herbicide application rates needed to control weed growth. This renders many herbicides unsuitable for controlling weeds in the presence of certain crops. Where weed growth in crops is uncontrolled however, this results in lower crop yield and reduced crop quality, as weeds will compete with crops for nutrients, light and water. Reduction in herbicidal injury to crops without an unacceptable reduction in the herbicidal action can be accomplished by use of crop protectants known as "safeners", also sometimes referred to as "antidotes" or "antagonists".

4-Benzoylisoxazoles are known to possess herbicidal properties, for example see European Patent Publication Nos. 0418175, 0487357, 0527036 and 0560482. European Patent Publication Nos. 0496630, 0496631, 0625505 and 0625508 disclose certain 1-phenyl-2-cyano-1,3-dione derivatives possessing herbicidal properties. European Patent Publication No. 0213892 discloses herbicidally active enols. These compounds possess very good levels of herbicidal activity, but at higher dose rates there can be a risk of crop phtyotoxicity.

The present invention seeks to provide compositions of these herbicides for use in combination with antidotes therefor for reduction of injury to crops, especially wheat, due to phytotoxicity of these herbicides at certain dose rates or under certain conditions of use.

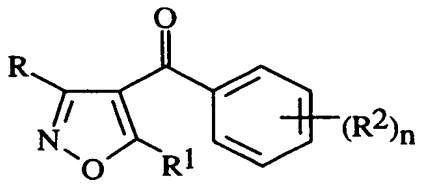
Description of the Invention

The invention provides a method of reducing phytotoxicity at a crop plant locus caused by at least one 4-benzoylisoxazole herbicide and/or 2-cyano-1,3-dione herbicide which comprises applying to the crop plant locus at least one sulfonylurea herbicide.

Suprisingly, the applicants have found that the presence of at least one sulfonylurea herbicide allows any crop phytotoxicity by the isoxazole and/or 2-cyano-1,3-dione herbicide to be reduced in the presence of at least one sulfonylurea herbicide, while maintaining a good level of weed control.

The method of the invention reduces phytotoxicity by a safening effect of the sulfonylurea. The invention also provides a method whereby the total amount of herbicide may be reduced by virtue of a synergistic effect.

Preferably the 4-benzoylisoxazole herbicide has the general formula (I):



(I)

wherein

R is hydrogen or $-CO_2R^3$;

R^1 is C_{1-6} alkyl or C_{3-6} cycloalkyl optionally bearing C_{1-6} alkyl;

R^2 is selected from halogen (e.g. chlorine or bromine), $-S(O)_pMe$ CH_2SO_qMe, C_{1-6} alkyl, C_{1-6} haloalkyl (e.g. CF_3), C_{1-6} alkoxy or C_{1-6} haloalkoxy;

n is two or three; p is zero, one or two; q is zero, one or two; and

R^3 is C_{1-4} alkyl.

In formula (I) above R^1 is preferably cyclopropyl.

In formula (I) above preferably one group R^2 represents $-S(O)_pMe$, most preferably in the 2-position of the benzoyl ring.

4-Benzoylisoxazoles of formula (I) above of particular interest in the method of the invention include the following:

A. 5-cyclopropyl-4-(2-methylsulfonyl-4-trifluoromethyl)benzoylisozaxole;

B. ethyl 5-cyclopropyl-4-(2-methylsulfonyl-4-trifluoromethyl)benzoylisoxazole-3-carboxylate;

C. ethyl 5-cyclopropyl-4-[3,4-dichloro-2-(methylsulphenyl) benzoyl]isoxazole-3-carboxylate;

D. 5-cyclopropyl-4-[4-bromo-2-(methylsulfonylmethyl)benzoylisoxazole;

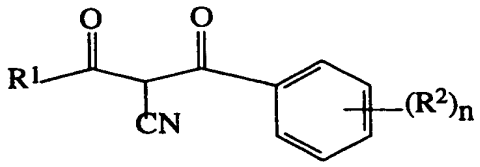
E. 5-cyclopropyl-4-(4-methylsulfonyl-2-trifluoromethyl)benzoylisoxazole; and

F. 5-cyclopropyl-4-(4-chloro-2-methylsulfonyl)benzoylisoxazole.

The letters A to F are assigned to these compounds for reference and identification hereafter.

Compounds A, B and C are preferred.

Preferably the 2-cyano-1,3-dione derivative has the formula (II):



(II)

wherein R^1, R^2 and n are as defined above.

Compounds of formula II above may exist in enolic tautomeric forms that may give rise to geometric isomers around the enolic double bond. Furthermore, in certain cases the groups R^1 and R^2 may give rise to stereoisomers and geometric isomers. All such forms and mixtures thereof are embraced by the present invention.

The most preferred compound of formula (II) above is 2-cyano-3-cyclopropyl-1-(2-methylsulfonyl-4-trifluoromethylphenyl)propan-1,3-dione.

Preferably the sulfonylurea herbicide has the general formula (III):

$$R_4SO_2NR_5C(O)NR_6R_7 \text{ (III)}$$

wherein:

R_4 is selected from substituted or unsubstituted phenyl, substituted or unsubstituted thienyl, substituted or unsubstituted pyridyl, and substituted or unsubstituted imidazopyridinyl;

R_5 and R_6 are independently C_{1-6} alkyl or hydrogen; or

R_7 is selected from substituted or unsubstituted triazine or substituted or unsubstituted diazine. Preferably the substituted phenyl, thienyl, pyridyl or imidazopyridinyl moieties are substituted by halogen, alkyl, alkoxy, alkylcarbonyl, alkoxycarbonyl, substituted or unsubstituted amido, alkylthio, alkylsulfenyl, and alkylsulfonyl. The triazine and diazine moieties are preferably substituted by alkoxy or alkyl.

In the above definition, alkyl and moieties comprising it generally contain from one to six carbon atoms and are optionally substituted by one or more atoms from group consisting of bromine, chlorine, fluorine and iodine.

Preferably the sulfonylurea herbicide has the general formula (III) with one or more of the following features:

R_4 is selected from 2-substituted phenyl, 2-substituted-3-thienyl, 3-substituted-2-pyridyl; 2-substituted-imidazo[1,2-a]pyridine;

R_5 and R_6 are independently methyl or hydrogen; and

R_7 is selected from 4,6-dimethoxy-1,3,5-triazine; 4-methoxy-6-methyl-1,3,5-triazine; 4,6-bisdifluoromethoxy-1,3-diazine, and 4,6-dimethoxy-1,3-diazine.

Even more preferably the sulfonylurea herbicide has the general formula (III) wherein:

R_4 is selected from 2-chlorophenyl; 2-methoxycarbonylphenyl, 2-haloalkylphenyl, 2-haloalkoxyphenyl, 2-methoxycarbonyl-3-thienyl, 3-dialkylamido-2-pyridyl and 2-alkylsulfonylimidazo[1,2-a]pyridine;

R_5 and R_6 are independently selected from hydrogen or methyl; and

R_7 is selected from 4,6-dimethoxy-1,3,5-triazine; 4-methoxy-6-methyl-1,3,5-triazine; 4,6-bisdifluoromethoxy-1,3-diazine; and 4,6-dimethoxy-1,3-diazine.

Preferably there are one or two sulfonylurea herbicides present selected from:

1-(2-chlorophenylsulfonyl)-3-(4-methoxy-6-methyl-1,3,5-triazin-2-yl)urea (chlorsulfuron);

2-[4-methoxy-6-methyl-1,3,5-triazin-2-yl(methyl)carbamoylsulfamoyl]benzoic acid (tribenuron-methyl);

3-(4-methoxy-6-methyl-1,3,5-triazin-2-ylcarbamoylsulfamoyl)thiophen-2-carboxylic acid (thifensulfuron methyl);

2-(4-methoxy-6-methyl-1,3,5-triazin-2-ylcarbamoylsulfamoyl)benzoic acid (metsulfuron);

1-[2-(2-chloroethoxy)phenylsulfonyl]-3-(4-methoxy-6-methyl-1,3,5-triazin-2-yl)urea (triasulfuron);

1-(4-methoxy-6-methyl-1,3,5-triazin-2-yl)-3-[2-(3,3,3-trifluoropropyl)-phenylsulfonyl]urea (CGA-152005 or prosulfuron);

1-(4,6-dimethoxypyrimidin-2-yl)-3-(3-dimethylcarbamoyl-2-pyridylsulfonyl)urea (nicosulfuron);

2-[4,6-bis(difluoromethoxy)pyrimidin-2-yl-carbamoylsulfamoyl]benzoic acid (primisulfuron);

N-[[(4,6-dimethoxy-2-pyrimidinyl)amino]carbonyl]-3-ethylsulfonyl-2-pyridinesulfonamide (rimsulfuron or DPX-E9636) and

1-(2-ethylsulfonylimidazo[1,2-a]pyridin-3-ylsulfonyl)-3-(4,6-dimethoxypyrimidin-2-yl)urea (MON 37500: CAS Registry Number 141776-32-1).

Sulfonylurea herbicides may be prepared by references found in The Pesticide Manual, 10th Edition, C. Tomlin editor; British Crop Protection Association, 1994, or by those known to the skilled addressee.

Most preferably there are two sulfonyl urea herbicides present. Preferably these are selected from thifensulfuron methyl, tribenuron methyl and MON 37500.

The amount of sulfonylurea herbicide used in the method of the invention varies according to a number of parameters including the weeds to be controlled, the crop to be protected, the amount and rate of herbicide applied, and the edaphic and climatic conditions prevailing. Also, the selection of the specific antidotes for use in the method of the invention, the manner in which it is to be applied and the determination of the activity which is non-phytotoxic but antidotally effective, can be readily performed in accordance with common practice in the art.

By "non-phytotoxic" is meant an amount of the antidote which causes at most minor or no injury to the desired crop species. By "antidotally effective" is meant an antidote used in an amount which is effective as an antidote to decrease the extent of injury caused by the herbicide to the desired crop species.

The dose rate of the benzoylisoxazole herbicide and/or 2-cyano-1,3-dione herbicide is generally from about 5 to about 500 grammes per hectare (g/ha), preferably from about 15 to about 200 g/ha, more preferably from about 20 to about 120 g/ha, even more preferably from about 70 to about 90 g/ha.

The dose rate of the sulfonylurea herbicide is generally from about 1 to about 250 g/ha, preferably from about 1 to about 100 g/ha, more preferably from about 3 to about 20 g/ha and even more preferably from about 5 to about 15 g/ha.

Preferably the weight ratio of isoxazole/dione:sulfonyurea herbicide is from about 500:1 to about 1:50, preferably from about 200:1 to about 1:7, more preferably from about 40:1 to about 1:1, and even more preferably from about 18:1 to about 5:1.

The method of the invention can be applied pre- or post-emergence of the crop. Where the crop is a cereal crop (such as wheat) the herbicide is preferably applied post-emergence of the crop species.

According to a further feature of the present invention there is provided a composition comprising:

(a) at least one 4-benzoylisoxazole herbicide or 2-cyano-1,3-dione herbicide; and

(b) at least one sulfonylurea herbicide,

in association with an agriculturally acceptable diluent or carrier and/or optionally a surface active agent. The composition may be provided as a ready-to-use formulation (e.g. where (a) and (b) are premixed), or may be formed as a tank mix in accordance with standard techniques in the art.

The herbicidal action of the composition may be substantially higher than the sum of the effects of the individual agents. The effect may be a synergistic effect.

The composition according to the invention can be used both in conventional methods of cultivation (strip cultivation with suitable strip width) and in plantation cultivation (e.g., vines, fruit, citrus), as well as in industrial plants and track systems, on roads and squares, but also to handle stubble and in the minimum-tillage method. They are also suitable as burners (for killing foliage, e.g. in potatoes) or as defoliants (e.g. in cotton). They are also suitable for use on fallow areas. Other areas of use are in tree nurseries, forests, grasslands, and int he cultivation of ornamental plants.

Examples of weeds which can be combatted well by the agents or combinations of agents according to the invention are:

Dicotyledon weeds of the genera sinapis, lepidium, galium, stellaria, matricaria, anthemis, galinsoga, chenopodium, urtica, senecio, amaranthus, portulaca, xanthium, convolvulus, ipomoca, polygonum, sesbania, ambrosia, solanum, cirsium, carduus, sonchus, rorippa, rotaia, lindernia, lamium, veronica, abutilon, emex. sida, datura, viola, galeopsis, papaver, centaurea, trifolium, ranunculus, taraxum, and mentha.

Monocotyledon weeds of the genera echinochloa, setaria, panicum, digitaria, phleum, poa, festuca, eleusine, brachiaria, lolium, bromus, avena, cyperus, sorghum, agropyron, cynodon, monochoria, fimbristylis, sagittaria, eleocharis, scirpus, papalum, ischaemum, spenoclea, dactyloctenium, agrostis, alopecurus, apera.

However, the use of the agents and combinations of agents according to the invention is in no way limited to these genera, but rather extends in the same way to other plants.

The crops that may be protected by the method of the invention include corn, rice, wheat, soya, sorghum and cotton. The method of the invention is preferably performed where the crop to be protected is wheat.

The invention also provides a product comprising at least one 4-benzoylisoxazole herbicide and/or 2-cyano-1,3-dione herbicide and a sulfonylurea herbicide for simultaneous, separate or sequential use in the control of weeds at a locus.

The following non-limiting Examples illustrate the invention.

Example 1

Compositions containing Compound A (formulated as a wettable powder containing 75 % active ingredient) alone, in tank-mixture with Triton Ag 98 (trademark, 0.25% v/v; a non-ionic surfactant) and in tank-mixture with Triton Ag 98, thifensulfuron methyl and tribenuron methyl were mixed together in a spray tank (the latter two sulfonylurea herbicides were used as the commercial

formulation HARMONY EXTRA™ a wettable powder containing *75%* combined active ingredients) were sprayed at a volume of 225 liters per hectare over Penawawa Soft White Spring Wheat which had emerged from seeding and grown to a height of about 18 cm. Comparative ratings for damage (by visual inspection of the degree of chlorosis present) to the wheat were taken at 4 and 8 days after treatment (DAT) in comparison with untreated control. The following results were observed:

		Percent Chlorosis	
Mixture	Rate Active Ingredient (g/ha)	4 DAT	8 DAT
Compound A	70	0	0
Compound A+ Triton AG 98	70	23	60
Compound A + Triton AG 98 thifensulfuron methyl tribenuron methyl	70 11.6 5.8	15	37

Example 2

The same procedure as described in Example 1 was followed replacing Compound A with Compound B (which was formulated as a suspension concentrate containing 4% active ingredient). The following results were obtained.

		Percent Chlorosis	
Mixture	Rate Active Ingredient (g/ha)	4 DAT	8 DAT
Compound B	85	0	0
Compound B + Triton Ag 98	85	18	38
Compound B + Triton Ag 98 thifensulfuron methyl tribenuron methyl	85 11.6 5.8	11	20

Example 3

Compound C as a 25% emulsifiable concentrate and prosulfuron (as a 20% wettable powder) were suspended in tap water and applied post-emergence both alone and in combination at a range of concentrations to Xanthium strumarium, Amaranthus retroflexus ,and Setaria faberi. Treatment effects were assessed visually 20 days after treatment. The percentage damage compared to untreated controls was recorded for each species.

The nature of the interaction between the two components was determined using the responses of the herbicides applied singly in calculating the expected response when they are combined (COLBY, S.R., "Calculating synergistic and antagonstic response of herbicide combinations". Weeds 15, pages 20-22, 1967):

$$We = X + \frac{Y \bullet (100 - X)}{100}$$

wherein

X = Percentage mortality, compared with untreated controls, after treatment with Compound C at a rate of application of p g/hectare.

Y = Percentage mortality, compared with untreated controls, after treatment with prosulfuron at a rate of application of q g/hectare.

We = The expected pesticidal activity (percentage mortality compared with untreated controls) after treatment with Compound C and prosulfuron at a rate of application of p + q g/ha.

In the tables that follow the figures used for weed control are percentages reduction in growth when compared with untreated controls. The figures in parentheses are those expected using the Colby formula.

Table 1

Post-emergence treatment of Xanthium strumarium with various mixtures of Compound C and prosulfuron

Prosulfuron

Cpd C	Dose g/ha	0	1	2	4	8
	0	-	0	15	0	55
	8	0	5 (0)	15 (15)	25 (0)	65 (55)
	16	5	25 (5)	40 (19)	60 (5)	90 (57)
	32	15	20 (15)	45 (28)	45 (15)	87 (62)
	63	20	30 (20)	35 (32)	62 (20)	77 (64)
	125	40	55 (40)	85 (49)	80 (40)	95 (73)

Table 2

Post-emergence treatment of Amaranthus retroflexus with various mixtures of Compound C and prosulfuron

Prosulfuron

Cpd C	Dose g/ha	0	4	8
	0	-	20	35
	8	20	60 (36)	77 (48)
	16	40	60 (52)	75 (61)
	32	52	82 (62)	87 (69)

Table 3

Post-emergence treatment of Setaria faberi with various mixtures of Compound C and prosulfuron

Prosulfuron

Cpd C	Dose g/ha	0	1	2	4	8	16
	0	-	0	5	20	20	32
	16	35	50 (35)	50 (38)	60 (48)	60 (48)	75 (56)

For a range of mixtures of Compound C with prosulfuron against Xanthium strumarium, Amaranthus retroflexus and Setaria faberi the observed response was greater than expected response thus indicating synergism.

While the invention has been described in terms of various preferred embodiments, the skilled artisan will appreciate that various modifications, substitutions, omissions, and changes may be made without departing from the spirit thereof. Accordingly, it is intended that the scope of the present invention be limited solely by the scope of the following claims, including equivalents thereof.

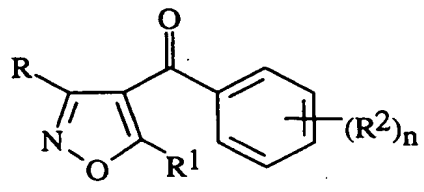
CLAIMS

1. A method of reducing phytotoxicity at a crop plant locus caused by at least one 4-benzoylisoxazole herbicide and/or 2-cyano-1,3-dione herbicide which comprises applying to the crop plant locus at least one sulfonylurea herbicide.

2. The method according to Claim 1 wherein phytotoxicity is reduced by a safening effect of the sulfonylurea.

3. The method according to Claim 1 or Claim 2 wherein the total amount of herbicide is reduced by virtue of a synergistic effect.

4. The method according to any one of the foregoing claims wherein the 4-benzoylisoxazole has the general formula (I):



(I)

wherein

R is hydrogen or $-CO_2R^3$;

R^1 is C_{1-6} alkyl or C_{3-6} cycloalkyl optionally bearing C_{1-6} alkyl;

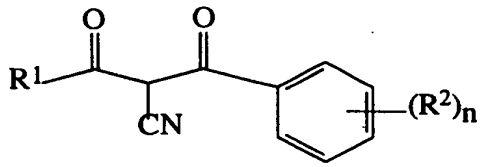
R^2 is selected from halogen, $-S(O)_pMe$, $CH_2S(O)_qMe$, C_{1-6} alkyl, C_{1-6} haloalkyl, C_{1-6} alkoxy and C_{1-6} haloalkoxy;

n is two or three; p is zero, one or two; q is zero, one or two; and

R^3 is C_{1-4} alkyl.

5. The method according to any one of the foregoing claims in which the 4-benzoylisoxazole is 5-cyclopropyl-4-(2-methylsulfonyl-4-trifluoromethyl)benzoylisozaxole.

6. The method according to any one of Claims 1 to 3 in which the 2-cyano-1,3-dione herbicide has the formula (II):



(II)

wherein

R^1 is C_{1-6} alkyl or C_{3-6} cycloalkyl optionally bearing C_{1-6} alkyl;

R^2 is selected from halogen, $-S(O)_pMe$ and C_{1-6} alkyl, C_{1-6} haloalkyl, C_{1-6} alkoxy and C_{1-6} haloalkoxy;

n is two or three; and p is zero, one or two.

7. The method according to any one of the foregoing claims in which the crop plant to be protected is corn, rice, wheat, soya, sorghum, and cotton; preferably wheat.

8. The method according to any one of the foregoing claims in which the dose rate of the benzoylisoxazole herbicide and/or 2-cyano-1,3-dione herbicide is from about 5 to about 500 g/ha.

9. The method according to any one of the foregoing claims wherein the sulfonylurea herbicide has the general formula (III):

$$R_4SO_2NR_5C(O)NR_6R_7 \text{ (III)}$$

wherein:

R_4 is selected from substituted or unsubstituted phenyl, substituted or unsubstituted thienyl, substituted or unsubstituted pyridyl, and substituted or unsubstituted imidazopyridine;

R_5 and R_6 are independently C_{1-6} alkyl or hydrogen; and

R_7 is selected from substituted or unsubstituted triazine or substituted or unsubstituted diazine.

10. The method according to Claim 9 wherein the sulfonylurea is:

1-(2-chlorophenylsulfonyl)-3-(4-methoxy-6-methyl-1,3,5-triazin-2-yl)urea;

2-[4-methoxy-6-methyl-1,3,5-triazin-2-yl(methyl)carbamoylsulfamoyl]benzoic acid;

3-(4-methoxy-6-methyl-1,3,5-triazin-2-ylcarbamoylsulfamoyl)thiophen-2-carboxylic acid;

2-(4-methoxy-6-methyl-1,3,5-triazin-2-ylcarbamoylsulfamoyl)benzoic acid;

1-[2-(2-chloroethoxy)phenylsulfonyl]-3-(4-methoxy-6-methyl-1,3,5-triazin-2-yl)urea;

1-(4-methoxy-6-methyl-1,3,5-triazin-2-yl)-3-[2-(3,3,3-trifluoropropyl)-phenylsulfonyl]urea;

1-(4,6-dimethoxypyrimidin-2-yl)-3-(3-dimethylcarbamoyl-2-pyridylsulfonyl)urea;

2-[4,6-bis(difluoromethoxy)pyrimidin-2-yl-carbamoylsulfamoyl]benzoic acid;

N-[[(4,6-dimethoxy-2-pyrimidinyl)amino]carbonyl]-3-ethylsulfonyl-2-pyridinesulfonamide; or

1-(2-ethylsulfonylimidazo[1,2-a]pyridin-3-ylsulfonyl)-3-(4,6-dimethoxypyrimidin-2-yl)urea.

11. A composition comprising:

(a) at least one 4-benzoylisoxazole herbicide or 2-cyano-1,3-dione herbicide; and

(b) at least one sulfonylurea herbicide in association with an agriculturally acceptable diluent or carrier.

12. The composition according to Claim 11 in which the 4-benzoylisoxazole is 5-cyclopropyl-4-(2-methylsulfonyl-4-trifluoromethyl)benzoylisozaxole.

13. A product comprising at least one 4-benzoylisoxazole herbicide and/or 2-cyano-1,3-dione herbicide and a sulfonylurea herbicide for simultaneous, separate or sequential use in the control of weeds at a locus.

INTERNATIONAL SEARCH REPORT

Inter onal Application No
PCT/EP 98/04006

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 A01N47/36 //(A01N47/36,43:80,41:10,37:42)

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 A01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 96 21357 A (ZENECA LTD) 18 July 1996	

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

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"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
21 October 1998	29/10/1998

Name and mailing address of the ISA	Authorized officer
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INTERNATIONAL SEARCH REPORT

...formation on patent family members

Inter ...nal Application No
PCT/EP 98/04006

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